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Synthesis and Characterization of NiO-ZnO Nano Composite

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ABSTRACT

A novel NiO-ZnO nanocomposite has been synthesized by a sol-gel method based on polymeric network of polyvinyl alcohol (PVA) . In this work mixed solvent of 50:50 ethanol water was used to dissolve zinc nitrate, nickel nitrate and PVA. The mixture was heated to 80^{O}C to form a homogeneous gel solution. The obtained gel was slowly heated to evaporate the solvent to form a hard homogeneous gel. The hard gel was calcinated at temperature 600^{O}C for 4 hours and 8 hours and converted into nanocomposites. The prepared nanocomposites have been characterized using X-Ray Diffraction (XRD), Transmission Electron Microscope, FTIR, UV-VIS. In the observed spectral features , the peak position, intensity and bandwidth were related to some structural properties of investigated samples. The size of nano particles of NiO-ZnO nano composites heated at 600^{O}C for 4 hours and 8 hours using Scherrer Formula comes out to be 8.5 nm and 17.1 nm. NiO-ZnO nanocomposites shows increase in absorption when it goes from UV region to visible region.

Keywords: Nano composite, XRD ,FTIR ,UV-VIS, SEM.

INTRODUCTION

ZnO nano particles are of great importance due to its low cost and other properties like good electrical, optical, nontoxic behaviour. It has many applications in different fields like solar cells, gas sensors, spintronics, ultraviolet lasers etc. (1-2).

NiO is the most exhaustively investigated transition metal oxide. It is NaCl-type antiferromagnetic oxide semiconductor. It offers promising candidature for many applications such as solar thermal absorber, catalyst for oxygen evolution, photo electrolysis and electro chromic device. Nickel oxide is also a well-studied material as the positive electrode in

batteries. NiO is a P-type semiconductor and its electrical conduction is almost entirely contributed from electron hole conduction . NiO is preferred for high electro chromic efficiency, low cost and high dynamic dispersion.

The study of composite material i.e mixture consisting of at least two phases of different chemical compositions has been of great interest from both fundamental and practical point of view. The physical properties of such materials can be combined to produce material of desired response. Composites have good potential for various industrial fields because of their excellent properties such as high hardness, high melting point, low density, low coefficient of thermal expansion, high thermal conductivity, good chemical stability and improved mechanical properties such as higher specific strength, better wear resistance and specific modulus.(3-4).

Composites are used in making solar cells, optoelectronic device elements, laser diodes and light emitting diodes (LED), industrial applications in aircraft, military and car industry. Besides this, the composites getting from the transition metal oxides are started to use as humidity and gas sensors. The use of these specialized materials can provide solution to difficult engineering problems. A chemical synthesis of organic-inorganic composites can be provided by the sol-gel process. The sol-gel process is broadly defined as one in which a useful product is prepared from a solution or suspension of precursor materials via hydrolysis and polycondensation.

EXPERIMENTAL

In this work mixed ethanol-water solvent (50:50) was used to dissolve 2 gm zinc nitrate, 2 gm nickel nitrate and 8 gm PVA, the mixture was heated to $800^{o}C$ to form a homogeneous sol solution. The obtained sol was slowly heated to evaporate the solvent and it form a hard homogeneous gel. The Pyrrolysis of the final gel was performed at a temperature of $600^{o}C$ for 4 hours and 8 hours. During the Pyrrolysis process, the PVA polymeric network through the outer surface, zinc and cadmium nitrate salts simultaneously calcinated and converted into NiO-ZnO nano composite. The obtained samples were crushed to prepare a fine powder.

RESULTS AND DISCUSSION

XRD

The XRD pattern of NIO-ZnO nanocomposites which were calcinated at $600^{0}C$ for 4 hours and 8 hours are shown in fig. 1a and fig. 1b respectively. Both the calcinated samples are polycrystalline in nature. The particle size of as prepared samples was found using Scherrer formula

$$d= 0.9\lambda/\beta\cos\theta$$

Where d= average particle size, β is full width at half maxima, θ is the Bragg angle, λ is the wavelength in angstrom. We use the most intense peak to calculate average crystalline size. It can be seen that average size of NiO-ZnO nanocomposites heated for 4 hours comes out to be 8.5nm and of sample heated for 8 hours comes out to be of

17.1nm.Thus it can be seen that the particle size increases with increase in time of calcinations (5-6).

SEM

SEM of NiO-ZnO nanocomposits calcinated at 600ºC at 4 h and 8 h is shown in fig. 2a and fig. 2b respectively. Scanning electron microscopy was performed in order to investigate the morphology of the nanocomposites. In Nanocomposite which were annealed at 600^{O}C for 4 h and for 8 h, it can be seen from the images that the particle size of nanocomposites are in the range of nanometers and having spherical and porous structure. The particles are uniformly distributed and attached to each other through the grain boundary to form agglomerated particles.(7-9)

UV-VIS

UV-VIS spectra of NiO-ZnO nanocomposite is shown in fig. 3a and fig. 3b. The UV-VIS spectra of NiO-ZnO when heated at 600^{O}C for 4 h is different from spectra when NiO-ZnO nanocomposite heated at 600^{O}C for 8 h. Two new peaks appeared at 312 nm and at 390 nm when heated for 8 h. Absorption with wavelength first decreases in the UV region and then increases in the VIS region in both samples when heated for 4 h and 8 h for 600^{O}C. Also absorption is more in samples heated for 4 h then in samples heated for 8 h at 600^{o}C. ZnO and NiO nanoparticles have better efficiency in UV region(200-380nm) and it drastically decrease in visible region whereas in NiO-ZnO nanocomposite the spectra shows increase in absorption when it goes from UV region to visible region. (10-12)

FTIR

FTIR spectra of NiO-ZnO nanocomposites when heated for 4h and 8h at 600^{O}C is shown in fig. 4a and fig. 4b respectively. FTIR spectra obtained when heated at 600 ^{0}C for 4 hours is different from FTIR spectra when heated at 600 ^{0}C for 8 hours. Four new peaks were found to be observed at 1630.71, 1470.50,710.59 and 510.00 cm^{-1}.

A broad band around 3400 cm^{-1} corresponds to stretching mode of OH group which is contributed by water contents. The peak around 2900cm^{-1} is due to C-H bond .The peak around 2300 cm^{-1} can be attributed due to presence of oxygen-oxygen bonds. Bands around 1600cm^{-1} can be contributed due to OH group in the metal alkoxides present in the gel. Band around 1450 cm^{-1} corresponds due to asymmetric stretching of C=O Bonds. Bands around 1100 cm^{-1} is due to C-O bonding. Band at 710.00 cm^{-1} may be due to Metal-Oxygen band. Bands at 619 cm^{-1} is ascribable due to intrinsic stretching vibration of metal oxide bond. Band observed at 619 cm^{-1} corresponds to stretching vibration of M-O-M, where M corresponds to metal occupying tetrahedral and octahedral (13-19).

The absorption bands appear to indicate the hydrophillic nature of the sol–gel synthesized material. Band around 490 cm^{-1} confirm the presence of crystalline NiO.

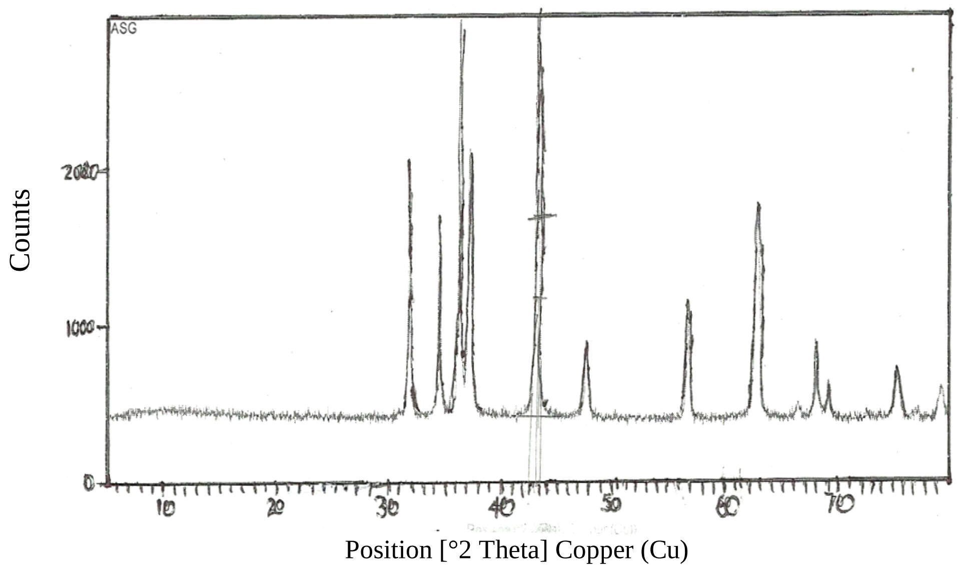

Fig 1a:- XRD spectra of NiO-ZnO nanocomposites annealed at 600^{O}C for 4 h.

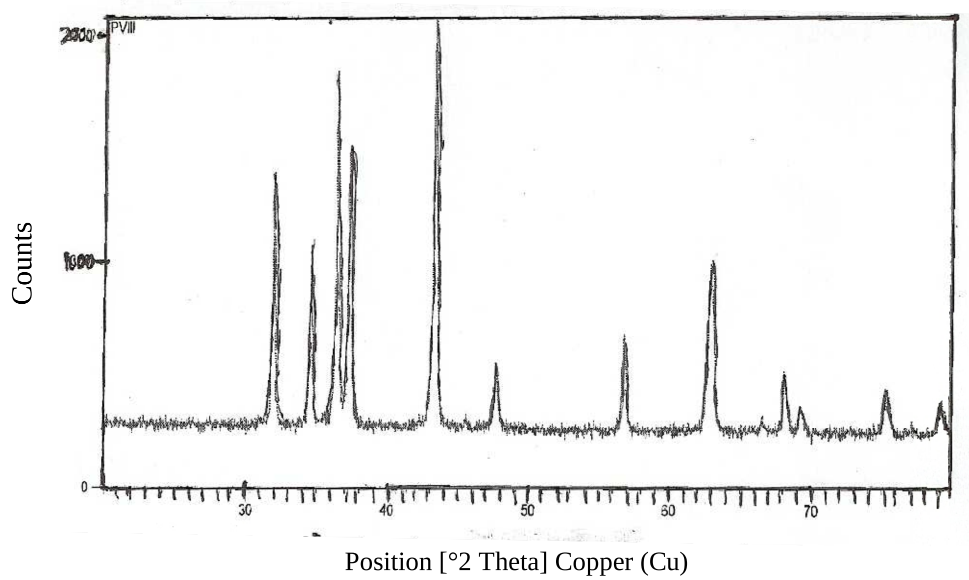

Fig 1b:- XRD spectra of NiO-ZnO nanocomposites annealed at 600^{O}C for 8 h.

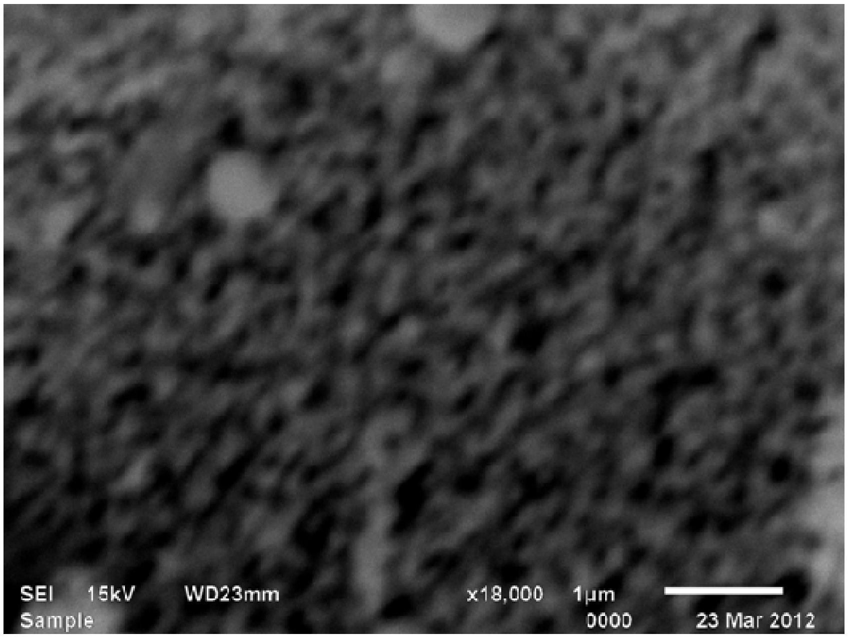

Fig 2a:-SEM spectra of NiO-ZnO nanocomposites annealed at 600^{O}C for 4 h.

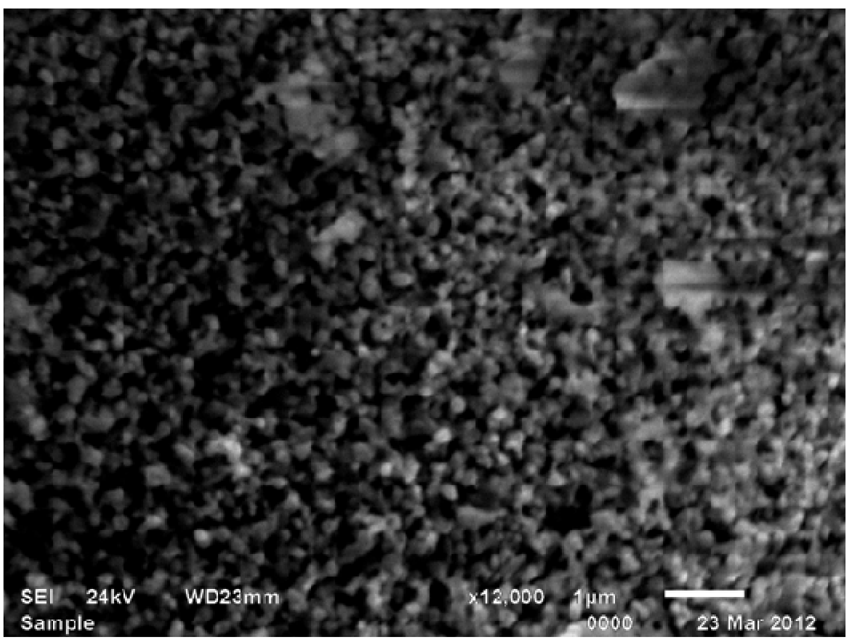

Fig 2b:- SEM spectra of NiO-ZnO nanocomposites annealed at 600^{O}C for 8 h.

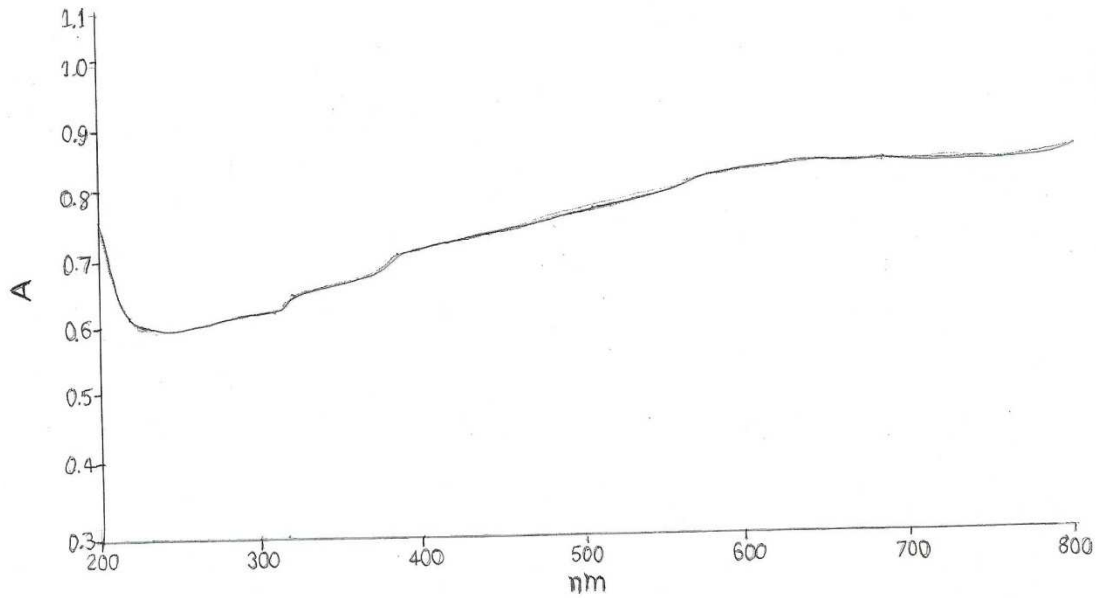

Fig 3a:- UV-VIS spectra of Nio-ZnO nanocomposite at 600^{O}C for 4 h.

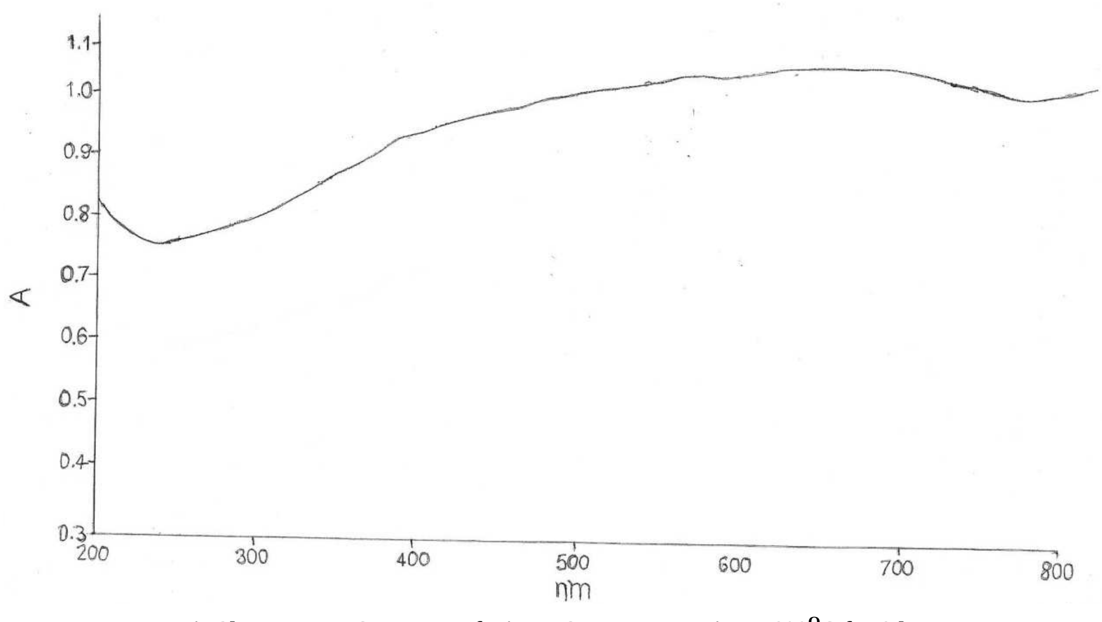

Fig 3b:- :- UV-VIS spectra of Nio-ZnO nanocomposite at 600^{O}C for 8 h.

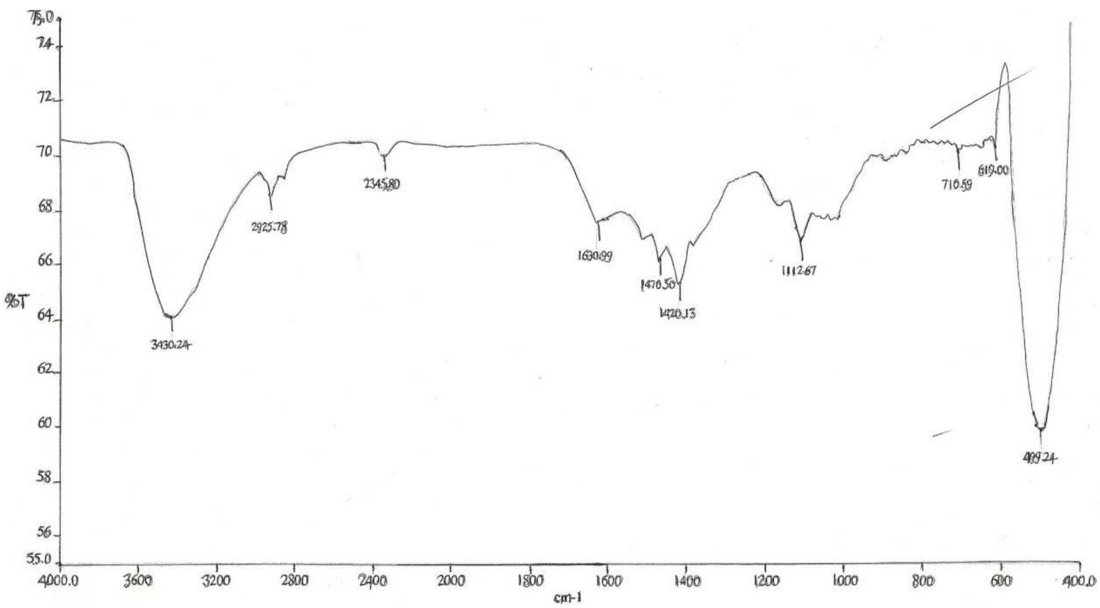

Fig 4a: FTIR spectra of NiO-ZnO nanocomposites annealed at $600^{O}C$ for 4 h.

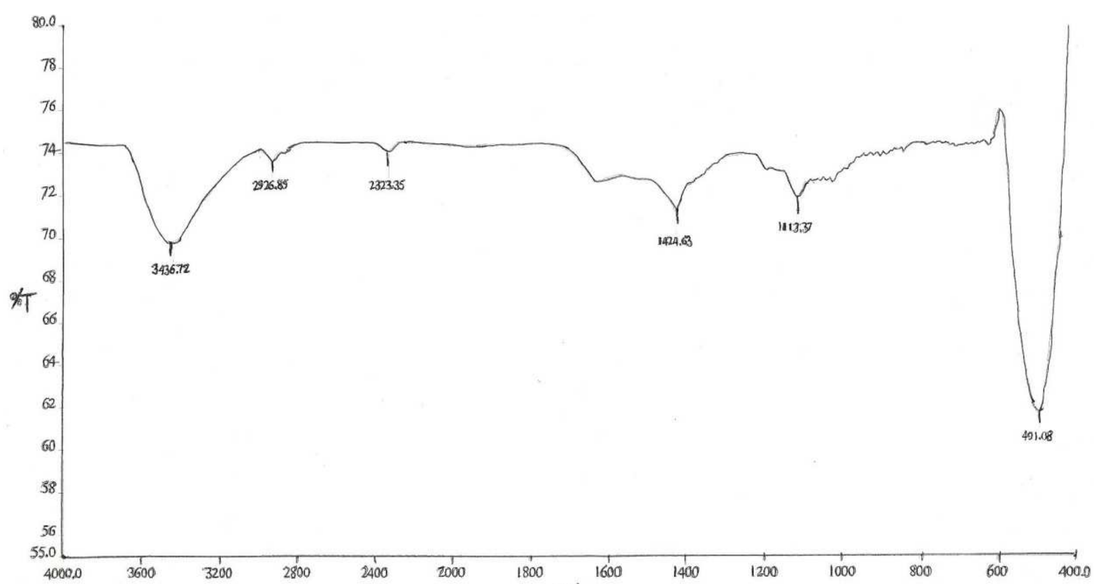

Fig 4b: FTIR spectra of NiO-ZnO nanocomposites annealed at $600^{O}C$ for 8 h.

CONCLUSION

The ZnO-NiO nanocomposites material have been prepared successfully by sol-gel method. The size of the nanocomposite so formed using XRD Scherrer formulae comes out to be 8.5 nm and 17.1 nm. The UV-VIS shows a increasing absorption in the visible region. The nano composites results were determined with XRD, UV-VIS, FTIR which confirm the formation of NiO-ZnO nanocomposites.

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